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भारतीय मानक

एल्यूमीनो फैरिक — विशिष्टि

(पाँचवा पुनरीक्षण)

Indian Standard

ALUMINO-FERRIC — SPECIFICATION

(Fifth Revision)

ICS 71.060.01

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Inorganic Chemicals Sectional Committee, CHD 1

FOREWORD

This Indian Standard (Fifth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1951 and was subsequently revised in 1962, 1975, 1980 and 1989. This standard is being revised again for improvement in the quality of drinking water. The requirement of insoluble mater has been reduced as the consumers of alum face the problem of choking of dosing lines in plants. Permissible limit of iron has also been reduced for product to be used in water treatment for drinking purposes; as higher iron content is associated with more impurities. Further, permissible limits of lead and arsenic have been decreased; and permissible limits of some heavy metals and organic impurities have been added based on the WHO guidelines for drinking water.

Improvement in the quality of potable water supply is a thrust area of all municipal and PHED water supply systems. As part of this quest to improve the quality of water supplied, there is a need for improved quality of alum (aluminium sulphate), used for water treatment for drinking purpose.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
ALUMINO-FERRIC — SPECIFICATION
(Fifth Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for alumino-ferric.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
264 : 2005	Specification for nitric acid (*third revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
1070 : 1992	Reagent grade water (*third revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
3025 (Part 28) : 1986	Methods of sampling and test (physical and chemical) for water and wastewater: Part 28 Sulphite (*first revision*)
3025 (Part 41) : 1992	Methods of sampling and test (physical and chemical) for water and wastewater: Part 41 Cadmium (*first revision*)
4905 : 1968	Methods for random sampling
11124 : 1984	Method for atomic absorption spectrophotometric determination of arsenic
12041 : 1987	Method for determination of mercury by atomic absorption spectrophotometry
12046 : 1987	Method for the determination of manganese by atomic absorption spectrophotometry
12074 : 1987	Method for determination of lead by atomic absorption spectrophotometry
13319 : 1992	Methods for determination of chromium by atomic absorption spectrophotometry — Test method
13320 : 1992	Determination of iron by atomic absorption spectrometry — Test method

3 GRADES

There shall be following grades of the material, namely;

a) Grade 1 and 2 in solid form;
b) Grade 3 in liquid form;
c) Grade 4 in solid form for treatment of water intended for drinking; and
d) Grade 5 in liquid form for treatment of water intended for drinking.

4 REQUIREMENTS

4.1 Description

The material shall essentially be aluminium sulphate octadecahydrate [$Al_2(SO_4)_3 . 18H_2O$]. The article of commerce usually contains 5 to 10 percent less water than the theoretical amount. It shall be in the form of lumps, coarse powder (kibbled) or slabs or in liquid form. The colour of the material shall be cream to brown for Grade 1, 2 and colourless to light yellow/brown for Grade 3 and white to off-white for Grade 4 and almost colourless for Grade 5.

4.2 The material, when tested according to the methods prescribed in Annex A, shall comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in bulk or in packages as agreed to between the purchaser and the supplier.

5.2 Marking

The packages shall be securely closed and marked with the following information:

a) Name and grade of the material (with the words 'Suitable for water purification for drinking purpose for Grades 4 and 5');
b) Name of the manufacturer and trade-mark, if any;
c) Mass of the material in package;
d) Date of manufacture; and
e) Batch number.

5.2.1 If the material is supplied in bulk, a card bearing all the information required as mentioned in **5.2** shall be placed with the supply.

Table 1 Requirement for Alumino-Ferric

(*Clauses* 4.2, 8.3, B-2.3.1 *and* B.2.5.2)

Sl No.	Characteristics	Requirement					Method of Test, Ref to Clause
		Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Insouble matter, percent by mass, *Max*	0.5	0.5	0.25	0.2	0.1	A-3
ii)	Soluble iron compound (as Fe), percent by mass, *Max*	0.7	0.7	0.35	0.04	0.02	A-4
iii)	Water soluble aluminium compounds (as Al_2O_3), percent by mass, *Min*	16	15	7.5	16	8	A-5
iv)	*p*H (of 5 percent aqueous solution), *Min*	2.7	2.7	2.7	2.7	2.7	A-6
v)	Free acidity (As H_2SO_4), Basicity (as Al_2O_3), percent by mass, *Max*	0.5	0.5	0.25	0.5	0.25	A-7
vi)	Lead (as Pb), parts per million, *Max*	—	—	—	20	10	A-8
vii)	Arsenic (as As_2O_3), parts per million, *Max*	—	—	—	4	2	A-9
viii)	Mercury (as Hg), parts per million, *Max*	—	—	—	0.4	0.2	A-10
ix)	Manganese (as Mn), parts per million, *Max*	—	—	—	20	10	A-11
x)	Chromium (as Cr), parts per million, *Max*	—	—	—	20	10	A-12
xi)	Cadmium (as Cd), parts per million, *Max*	—	—	—	4	2	A-13
xii)	Selenium (as Se), parts per million, *Max*	—	—	—	4	2	A-14
xiii)	Organic impurities:						
	a) Phenolic compounds (as C_6H_5OH), parts per million, *Max*	—	—	—	0.4	0.2	A-15
	b) Anionic detergents (as MBAS), parts per million, *Max*	—	—	—	80	40	A-16

5.2.2 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

5.2.3 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the license for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The method of drawing representative samples of the material from a lot, the number of tests to be performed and the method of finding the criteria of conformity of the material to the requirements of this standard shall be as prescribed in Annex B. In case of liquid alum, it is supplied in tankers and from the point of loading to unloading the tanker, the time gap is about 10 to 12 h or more. During that period, the insoluble settle down and many times the purchaser draws spot sample from the bottom which can give high value of insoluble matter. Hence, proper mixing should be done before drawing a representative sample.

ANNEX A

(*Clause* 4.2)

METHODS OF TEST FOR ALUMINO-FERRIC

A-1 QUALITY OF REAGENTS

Unless specified otherwise, A.R. grade chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 PREPARED SAMPLE

A-2.1 Procedure

If the material is in solid form, crush about 200 g of the material rapidly to pass through 1.00 mm IS Sieve. Keep it in a clean glass stoppered weighing bottle. Use this prepared sample for tests.

A-3 DETERMINATION OF INSOLUBLE MATTER

A-3.1 Reagents

A-3.1.1 *Dilute Sulphuric Acid* — Approximately 1 *N*.

A-3.2 Procedure

Weigh accurately about 10 g of the sample (for liquid alum, 20 g of the sample), mix with 10 ml of dilute sulphuric acid and dissolve in 100 ml of water by stirring and warming, if necessary. Filter through a tared filter paper preferably Whatman 41 or equivalent or a sintered glass crucible (G 4). Wash the residue once with hot water slightly acidulated with sulphuric acid and then with hot water till it is free from soluble compounds, and dry to constant mass at 105 to 110°C.

A-3.3 Calculation

$$\text{Insoluble matter, percent by mass} = \frac{100\,m}{M}$$

where

m = mass of the residue, in g; and

M = mass of the sample taken for the test, in g.

A-4 DETERMINATION OF SOLUBLE IRON COMPOUNDS

A-4.1 Test Method of Grades 1, 2 and 3

A-4.1.1 *Reagents*

A-4.1.1.1 *Concentrated hydrochloric acid* — See IS 265.

A-4.1.1.2 *Stannous chloride solution* — Dissolve 6.0 g of stannous chloride crystals ($SnCl_2.2H_2O$) in 60 ml of concentrated hydrochloric acid and dilute with distilled water to 100 ml. Keep this solution in a stoppered bottle.

A-4.1.1.3 *Mercuric chloride solution* — Saturated.

A-4.1.1.4 *Sodium diphenylamine sulphonate indicator solution* — Dissolve 0.5 g of sodium diphenylamine sulphonate in 100 ml of water.

A-4.1.1.5 *Standard potassium dichromate solution* — 0.05 *N*.

A-4.1.1.6 *Phosphoric acid*

A-4.1.2 *Procedure*

Weigh accurately about 10 g of the sample (for liquid alum, 20 g of the sample) and dissolve it in 50 ml of water and filter. To the filtrate, add about 10 ml of concentrated hydrochloric acid and heat to boiling. Reduce the iron by adding stannous chloride solution until the yellow colour of ferric chloride disappears. Do not add more than 2 to 3 drops of stannous chloride in excess. Cool the solution, add rapidly 10 ml of mercuric chloride solution and stir so that a little white precipitate of mercurous chloride forms slowly, indicating that a slight excess of stannous chloride was present. Allow to stand for 5 min. Add 5 ml of phosphoric acid and dilute to 200-250 ml with water. Add 5 drops of diphenylamine indicator and titrate with standard potassium dichromate solution until the colour of the solution changes to a deep blue which does not fade on stirring.

A-4.1.3 *Calculation*

$$\begin{array}{l}\text{Soluble iron compounds}\\ \text{(as Fe), percent by mass}\end{array} = \frac{5.584 \times V \times N}{M}$$

where

V = volume of standard potassium dichromate solution required for the titration, in ml;

N = normality of standard potassium dichromate solution; and

M = mass of the sample taken for the test, in g.

A-4.2 Test Method for Grades 4 and 5

A-4.2.1 *Principle*

Iron reacts with thiocyanate to give a series of internally red coloured compounds which remains in true solution. Strong acids (hydrochloric acid) should be present to compress the hydrolysis. Alternatively iron may be determined by atomic absorption spectrophotometer (Referee method).

A-4.2.2 *Reagents*

A-4.2.2.1 *Concentrated hydrochloric acid* (*35-37 percent*) — A.R. Grade.

A-4.2.2.2 *Potassium permanganate solution* — Dissolve 2 g of potassium permanganate solution in 1 000 ml of distilled water.

A-4.2.2.3 *Ammonium thiocyanate solution* — Dissolve 10 g of ammonium thiocyanate in 100 ml of distilled water.

A-4.2.2.4 *Standard iron solution 1ml = 0.1 mg Fe* — Dissolve 0.702 2g of A.R. Grade ferrous ammonium sulphate hexahydrate (Molecular weight 392.14 g) in 100 ml of water, add 5 ml of dilute sulphuric acid and run in continuously a dilute solution of potassium permanganate (2g/l) until a slight pink colour remains after stirring well. Dilute it up to 1 litre and mix thoroughly.

A-4.2.2.5 *Dilute sulphuric acid* — 1 : 5

A-4.2.3 *Procedure*

Weigh accurately to the nearest milligram about 0.5 g of solid alum (or 1 g of liquid alum) sample in a 100 ml beaker, add 5 ml of concentrated hydrochloric acid, warm it and then cool it to room temperature. Transfer it into 100 ml Nessler's cylinder. Take distilled water in another Nessler's cylinder as a blank. Add 5 ml of concentrated hydrochloric acid and 3-4 ml of potassium permanganate solution, followed by addition of 10 ml of ammonium thiocyanate to both the cylinders and shake. Add distilled water up to the mark. Shake well. Using a micro-pipette, add standard iron solution (1 ml = 0.1 mg Fe) to the blank till colour matches with that of sample in Nessler cylinder.

A-4.2.4 *Calculation*

$$\text{Soluble iron compound as Fe, percent by mass} = \frac{V \times 0.01}{M}$$

where

V = volume of standard iron solution, in ml; and

M = mass of sample taken for the test, in g.

A-4.3 Referee Method

By Atomic Absorption Spectrophotometer (AAS); follow the method as prescribed in IS 13320. Alternately, it can also be analyzed by ICP-OES.

A-5 DETERMINATION OF WATER SOLUBLE ALUMINIUM COMPOUNDS

A-5.1 Principle

Total aluminium in sample solution is converted to Al^{3+} ions by addition of dilute nitric acid and heating, followed by complexation of Al^{3+} with an excess of ethylenediamine tetra acetic acid disodium (EDTA) solution. Excess EDTA is titrated with a standard volumetric solution of zinc in the presence of xylenol orange as indicator.

$$Al^{3+} + EDTA^{4-} \longrightarrow (Al\ EDTA)^{-}$$

$$Zn^{2+} + EDTA^{4-} \longrightarrow (Zn\ EDTA)^{2-}$$

A-5.1.1 *Reagents*

A-5.1.1.1 *Nitric acid (1 : 12)* (*see* IS 264)

A-5.1.1.2 *M/20 EDTA* — Dissolve about 19 gm of ethylenediamine tetra acetic acid disodium salt dihydrate in distilled water, transfer to a 1 000 ml-volumetric flask and make up to the mark by distilled water and shake well.

A-5.1.1.3 *Sodium acetate buffer solution* — Dissolve 272 g of sodium acetate trihydrate in water and dilute to 1 litre.

A-5.1.1.4 *Xylenol orange indicator solution, 0.1 percent w/v* — Dissolve 0.1 g of xylenol orange powder in some water, shake and make up to 100 ml. Shake well.

A-5.1.1.5 *Hydrochloric acid* — A.R. Grade (*see* IS 265).

A-5.1.1.6 *M/50 zinc solution* — Weigh accurately about 1.308 g of A.R. Grade zinc metal pellets/granule, transfer to a 250 ml beaker, add small amount of water and 6-7 ml of concentrated hydrochloric acid A.R., heat to dissolve zinc, evaporate on a water bath nearly to dryness, dissolve the residue in water, transfer the contents quantitatively to a 1 000 ml-volumetric flask and make up to the mark.

$$\text{Molarity of zinc solution, } M = \frac{m}{65.37}$$

m = mass of zinc metal taken for preparation of solution, in g.

A-5.1.2 *Procedure*

Weigh accurately, to the nearest 1 mg, *S*g (for solid about 5 g, for liquid about 10 g) of the test sample in a weighing bottle. Transfer it quantitatively to a 500 ml-volumetric flask with aid of about 150 ml water, dissolve by shaking and make up to the mark.

Pipette out accurately 20 ml of this solution to a 250 ml-conical flask and add 2 ml of nitric acid (1:12), boil for 1 min, allow to cool and add 20 ml of *M*/ 20 EDTA solution exactly by pipette.

Adjust to *p*H 3 with nitric acid or 10 percent sodium hydroxide solution using *p*H meter or by thymol blue *p*H test paper. Boil for about 2 min.

Cool and add approximately 10 ml sodium acetate buffer solution (*p*H of mixture should be between 5-6) and 2-5 drops of xylenol orange solution (0.1 percent *w/v*).

Titrate the solution with *M*/50 zinc solution, making the end point, the time where the colour of the solution turns from pale yellow to pale red.

In parallel, pipette out 20 ml *M*/20 EDTA, add 2 ml of nitric acid (1:12) solution to 250 ml-conical flask with about 20 ml distilled water and proceed the blank test as described in **A-5.1.2**.

A-5.1.3 *Calculation*

$$\text{Water souble aluminium compounds as } Al_2O_3\text{, percent by mass} = \frac{127.45 \times M\ (d - a)}{S} - 1.429\,7\,A$$

where

M = molarity of standard zinc solution;

d = titre of standard zinc solution used in blank determination, in ml;

a = titre of standard zinc solution used for sample solution, in ml;

S = sample taken for test, in g; and

A = percent by mass of iron as determined in **A-4**.

A-6 *p*H

A-6.1 Procedure

Dissolve 5 g of the sample (10 g for liquid alum) in 100 ml of water and determine *p*H of the solution at 27°C ± 2°C with the help of a suitable *p*H meter, using glass electrode.

A-7 FREE ACIDITY/BASICITY

A-7.1 Reagents

A-7.1.1 *Dilute Sulphuric Acid* — 0.5 *N* approximately.

A-7.1.2 *Potassium Fluoride Solution* — Relative density approximately 1.32, neutralized with potassium hydroxide if acidic or with concentrated sulphuric acid if alkaline, using phenolphthalein solution as indicator.

A-7.1.3 *Phenolphthalein Indicator Solution* — 0.1 g in 100 ml of 60 percent rectified spirit.

A-7.1.4 *Standard Sodium Hydroxide Solution* — 0.5 *N*.

A-7.2 Procedure

Weigh accurately about 6 g of the sample and dissolve in 20 ml of carbon dioxide free water and 10 ml of dilute sulphuric acid. Cool the solution to 20°C and add 20 ml of potassium fluoride solution and 0.5 ml of phenolphthalein indicator. Titrate the solution against standard sodium hydroxide solution (V_1) till the end point which is indicated by a pink colour persisting for a minute.

A-7.2.1 Carry out a blank titration without the sample (V_2). If V_1 is less than V_2, the sample is basic and if V_2 is less than V_1, the sample is acidic.

A-7.3 Calculation

$$\text{Basicity (as } Al_2O_3\text{) percent by mass} = \frac{(V_2 - V_1) \times N \times E_1}{10\,M}$$

$$\text{Acidity (as } H_2SO_4\text{) percent by mass} = \frac{(V_1 - V_2) \times N \times E_2}{10\,M}$$

where

V_1 = volume of standard sodium hydroxide solution used in the titration of the material, in ml;

V_2 = volume of standard sodium hydroxide solution used in the blank titration, in ml;

N = normality of standard sodium hydroxide solution;

E_1 = equivalent mass of Al_2O_3;

E_2 = equivalent mass of H_2SO_4; and

M = mass of the sample taken for the test, in g.

A-8 DETERMINATION OF LEAD

Lead is determined by comparing the colour produced by the material with hydrogen sulphide against that produced by a standard lead solution.

A-8.1 Apparatus — Nessler cylinder, 100 ml capacity.

A-8.2 Reagents

A-8.2.1 *Standard Lead Solution* — Dissolve 1.6 g lead nitrate in water and make up the volume to 1 000 ml. Transfer exactly 10 ml of the solution to volumetric flask and dilute it again with distilled water to 1 000 ml mark. One milliliter of this solution contains 0.01mg of lead (as Pb). The dilute solution shall be freshly prepared.

A-8.2.2 *Acetic Acid N (Approximately)*

A-8.2.3 *Hydrogen Sulphide Gas*

A-8.3 Procedure

Weigh accurately 1 g of sample and dissolve it in 40 ml of water and transfer to a 100 ml Nessler cylinder. Add 5 ml of acetic acid. Pass hydrogen sulphide gas into the solution till colour is developed. Continue the passing of hydrogen sulphide gas till further no more colour develops. In the second Nessler cylinder, carry out a control test using 2 ml of standard lead solution in case of Grade 4 and 1ml for Grade 5 in place of the sample and same quantities of the other reagents. Dilute the content of each cylinder to 100 ml and shake well. Compare the colour produced in the two cylinders.

The limit prescribed in Table 1 shall be taken as not having been exceeded, if the intensity of colour produced with the material is not greater than that produced in the control test.

A-8.4 Referee Method

Determine lead content by atomic absorption spectrophotometer in accorance with method given in IS 12074. Alternatively it can be analyzed by ICP-OES.

A-9 ARSENIC

A- 9.1 Reagents

A-9.1.1 *Concentrated Hydrochloric Acid* — See IS 265.

A-9.1.2 *Brominated Hydrochloric Acid* — Shake 1 ml of bromine with 100 ml of concentrated hydrochloric acid.

A-9.1.3 *Stannous Chloride Solution* — Dissolve 80 g of stannous chloride in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

A-9.2 Procedure

Dissolve 1.0 g of the sample (for liquid alum, 2.0 g of the sample) in 6 ml of brominated hydrochloric acid and 20 ml of water and remove the excess of bromine with a few drops of stannous chloride solution. Add water to make up the volume to about 50 ml. Carry out the test for arsenic either by modified Gutzeit method or by silver diethyl dithiocarbamate method as prescribed in **5.1** and **5.2** of IS 2088 using for comparison 0.006 mg of arsenic trioxide (as As_2O_3).

A-9.3 Referee Method

By atomic absorption spectrophotometer (AAS), follow the method as prescribed in IS 11124. Alternatively, it can be analyzed by ICP-OES

A-10 DETERMINATION OF MERCURY

A-10.1 Mercury Analyzer Method

A-10.1.1 *Outline of the Method*

Mercury analyzer works on the principle of cold vapour atomic absorption spectrometry technique. Mercury ions are reduced to elemental state by stannous chloride and the solution is stirred vigorously so that an equilibrium is achieved between the mercury in the solution and air phase. The vapour is then purged into the absorption which is located in the light path of spectrometer.

As the cold vapour absorption technique of mercury is based on absorption of UV radiation by mercury atoms, all substances which absorb UV radiation will cause the positive error. Vapours of organic compounds like alcohols, ketones, esters and acids and water also absorb UV radiation. These can be avoided with suitable traps provided along with the instrument.

A-10.2 Apparatus

A-10.2.1 *Mercury Analyzer*, based on cold vapour atomic absorption spectrometry technique.

A-10.3 Reagents

A-10.3.1 *Nitric Acid* — 10 percent and 2 percent (*v/v)* (*see* IS 264).

A-10.3.2 *Concentrated Hydrochloric Acid* — A.R. Grade (*see* IS 265).

A-10.3.3 *Stannous Chloride* — 20 percent(*w*/*v*) in 10 percent hydrochloric acid (*v/v*) — Take 20 g stannous chloride in a clean beaker, add 10 ml of concentrated hydrochloric acid and dissolve while warming it over a hot plate. Boil for one minute, cool and dilute with distilled water to make 100 ml. Add 1-2 g of tin metal (pellet) after the preparation of the solution.

A-10.3.4 *Potassium Permanganate* — 4 percent (*w/v*) in 10 percent sulphuric acid — Dissolve 20 g of potassium permanganate in water and add carefully 50 ml of concentrated sulphuric acid. Make up volume to 500 ml using water.

A-10.3.5 *Hydroxylamine Hydrochloride* — 10 percent (*w/v*).

A-10.3.6 *Mercuric Chloride*, A.R. Grade.

A-10.3.7 *Potassium Dichromate*, 1 percent (*w/v*).

A-10.4 Preparation of Standard Graph

A-10.4.1 *Preparation of Stock Solution* — Dissolve 0.135 4 g of mercuric chloride A.R. Grade in 25 ml of 2 percent nitric acid (*v/v*). Add 1 ml of potassium dichromate solution of 1 percent (*w/v*) and make up to 100 ml with 2 percent nitric acid (*v/v*). 1 ml of this stock solution = 1 mg of Hg.

A-10.4.2 *Preparation of Standard Solution*

Take 10 ml of the above stock solution into a 1 000 ml-volumetric flask, add 10 ml of potassium dichromate solution 1percent (*w/v*) and make up to the mark with 2 percent nitric acid (*v/v*). 1 ml of this solution = 10 µg of Hg. Further dilute 10 ml of this (1 ml = 10 microgram) solution to 1 000 ml, using 10 ml of potassium dichromate solution 1percent (*w/v*) and make up to the mark with 2 percent nitric acid (*v/v*), which will contain 1 ml = 10 nanogram of Hg. This standard solution is generally stable for a period of at least a month.

A-10.4.3 *Procedure for Standard Graph*

By using the standard solution find out the optical density for 10, 20, 40, 60, 80, 100, 120, 140, 150, nanograms of mercury. By following the procedure described below, plot a standard curve indicating mercury in nanograms against optical density.

A-10.5 Procedure

A-10.5.1 *Blank Preparation*

Transfer 8 ml of 10 percent nitric acid solution and 2 ml of stannous chloride solution to the reaction vessel. Stir it for 5 min. Stop the stirring and take the reading for the mercury content, if any in the blank, repeat the stirring of the blank solution until no deflection/ reading is observed. Take various readings for the various known concentration of mercury in the aliquots and prepare the graph.

A-10.5.2 Weigh accurately about 5 g of the sample in a 50 ml-beaker, dissolve the sample in 25 ml-distilled water and transfer to a 250 ml-volumetric flask with the help of more distilled water. Add drop wise potassium permanganate 4 percent *w/v* till purple colour remains, shake well and add 2-3 drops of concentrated nitric acid, mix thoroughly and keep it for 5-10 min. Decolourize the solution by adding drop wise hydroxylamine hydrochloride solution 10 percent (*w/v*), shake well and make up to the mark with distilled water. Prepare a fresh blank (*see* **A-10.5.1**), stir it for 5 min, stop the stirring take reading for the mercury content, if any in the blank. Transfer 5 ml of the sample solution to the reaction vessel containing blank and determine the concentration of the mercury with the help of the standard graph (*see* **A-10.4.3**). Prepare a blank solution using all reagents in same quantities except sample in a 250 ml-volumetric flask, use distilled water in place of sample. Make up to mark with distilled water and shake well. Transfer 5 ml of this solution to reaction vessel containing blank in the same way as sample, due to presence of any mercury content in reagents which shall be used in analysis. Calculate mercury content from standard graph and subtract mercury content of this blank solution from sample result, and take for calculation of mercury content in sample.

A-10.6 Calculation

$$\text{Mercury (as Hg), ppm} = \frac{Y \times 50 \times 1\,000}{W}$$

where

Y = mg of mercury as Hg in 5 ml; and

W = mass of sample.

A-10.7 Referee Method

By atomic absorption spectrophotometer (AAS), follow the method as prescribed in IS 12041.

A-11 DETERMINATION OF MANGANESE

A-11.1 Principle

Manganese is oxidized to permanganate, violet colour of which is compared with standard visually.

A-11.2 Apparatus

A-11.2.1 *Nessler Cylinder*, 100 ml capacity.

A-11.3 Reagents

A-11.3.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*) (*see* IS 264).

A-11.3.2 *Dilute Phosphoric Acid* — 1 : 1 (*v/v*).

A-11.3.3 *Potassium Periodate* — A.R. Grade powder.

A-11.3.4 *Concentrated Sulphuric Acid* — A.R. Grade.

A-11.3.5 *Standard Maganese Solution* — Dissolve 0.307 6 g of manganese sulphate monohydrate ($MnSO_4.H_2O$) in water, add 1 ml of concentrated sulphuric acid A.R. Grade, and make up the volume to 1 000 ml in a volumetric flask. Dilute to 10 ml of this solution to 1 000 ml. One millilitre of this solution is equivalent to 0.001 mg of manganese (as Mn).

A-11.4 Procedure

Weigh accurately 1 g of solid sample (2 g of liquid sample) and dissolve in 100 ml beaker, add 30 ml of water and 5 ml of dilute nitric acid, heat to boil. Add 10 ml of dilute phosphoric acid and 0.6 to 0.8 g of potassium periodate. Again heat the solution to boiling and then boil for 10 min. Cool the solution to room temperature. Transfer the solution completely to a Nessler cylinder, make up the volume to 100 ml mark. Carry out a control test using 20 ml of standard manganese solution in place of the material and follow the procedure as mentioned above.

A-11.5 The material shall have be taken to have passed the test if the intensity of colour produced with the material is not greater than that of produced in the control test.

A-11.6 Referee Method

Determine manganese content by atomic absorption spectrophotometer in accordance with method given in IS 12046. Alternatively, it can be analyzed by ICP-OES.

A-12 DETERMINATION OF CHROMIUM

A-12.1 Principle

Chromium hexavalent is reduced to chromium trivalent by boiling with potassium iodide and sodium sulphite and co-precipitate with aluminium hydroxide. The aluminium hydroxide is dissolved in 1:1 sulphuric acid, and chromium is oxidized to hexavalent form by bromine water. Chromium is determined colourimetrically by reacting with diphenyl carbazide reagent, which gives a purple colour with chromium.

A-12.2 Reagents

A-12.2.1 *Sulphuric Acid* — 1 : 1 (*v/v*) and 1 : 9 (*v/v*).

A-12.2.2 *Phosphoric Acid* — 1 : 5 (*v/v*).

A-12.2.3 *Diphenyl Carbazide Reagent* — Dissolve 0.25 g of diphenyl carbazide and 0.4 g of phthalic anhydride in 100 ml ethanol. The solution is stable for one month.

A-12.2.4 *Bromine Water*

A-12.2.5 *Potassium Iodide Solution* — 16 percent (*w/v*).

A-12.2.6 *Sodium Hydroxide Solution* — 30 percent (*w/v*).

A-12.2.7 *Neutral Sodium Sulphite Solution*

A-12.2.8 *Potassium Dichromate* — A.R. Grade.

A-12.2.9 *Concentrated Hydrochloric Acid* — A.R. Grade.

A-12.2.10 *Aluminium Solution* — Dissolve 1 g of aluminium metal in a little quantity of A.R. Grade hydrochloric acid and dilute, transfer to 1 000 ml volumetric flask and make up to the mark with distilled water. Shake well.

A-12.3 Procedure

Weigh accurately 1 g of the solid sample (2 g liquid sample) in 100 ml-beaker, dissolve in water and transfer in 500 ml-volumetric flask, make up to the mark with water.

Pipette out 100 ml of sample and transfer it to a 1 000 ml-beaker, add 20 ml of potassium iodide and 20 ml of sulphuric acid and 100 ml of water. Boil for about 10 min. Add sodium sulphite in small portion, to decolourize the sample. In case the solution turns brown on further boiling continue adding sodium sulphite solution till the decolourization is stable and brown colour of iodine does not appear on boiling. Add sodium hydroxide drop wise, stir the solution well and adjust the *p*H to 6.0 to 6.5. Allow to settle the complete precipitate. Siphon out the clear solution and collect the aluminium precipitate in a small beaker. Filter through a sintered glass crucible and wash once with water. Dissolve the precipitate by pouring 20 ml of boiling sulphuric acid (1:9) into the crucible, apply suction after 2 to 3 min and again wash with hot water. Collect the filtrate in 250 ml- beaker. Add 4 ml of bromine water and stir well. Decolourize the yellow colour with a few drops of sodium hydroxide solution and add 1 ml excess. Boil for 5 min and add 1:1 sulphuric acid until the brownish colour appears. Add 2 ml of the acid in excess and boil again till brownish colour disappears. Cool and add 1 ml of phosphoric acid. Add 1ml of diphenyl carbazide solution and make up the solution to 100 ml, shake well. Wait for 10 to 15 min for full colour to develop and then find out the percentage transmittance on a spectrometer using 460 nm green filter and calculate the corresponding optical density. From the standard graph find out the concentration of chromium.

A-12.4 Preparation of Standard Graph

Weigh 0.283 g of dried potassium dichromate, dissolve in a little distilled water and dilute to 1 litre. One millilitre of this solution is equivalent to 0.1 mg of chromium (as Cr^{6+}). Further dilute 10 ml of the above solution to 1 litre. One millilitre of the diluted solution is equivalent to 1 µg of chromium. Take various volumes of the standard solution so as to give 5,10,15,20 and 25 µg of chromium. Add 15 ml of aluminium solution to each, dilute to about 50 ml and add 30 ml 1:1 sulphuric acid and 2 ml of bromine water. Further add 30 percent sodium hydroxide drop wise till the colour disappears and add 2 drops in excess. Boil for 5 min and cautiously add 1:1 sulphuric acid till the bromine colour reappears and add 0.5 ml acid in excess. Again boil for about 20 min until bromine colour disappears. Check for bromine, cool and add 1 ml of diphenyl carbazide solution and make up the volume to 100 ml. Let it stand for 15 min and then take the reading at the wave length of 460 nm. Find out corresponding optical density. For adjusting 100 percent transmittance use 50 ml water in place of standard solution and proceed in same manner as above. Plot the optical densities against the corresponding chromium concentrations.

A-12.5 Calculation

$$\text{Chromium as Cr, ppm} = \frac{\text{Mass, in microgram, of chromium in 100 ml} \times 5}{\text{Mass of sample taken for the test, in g}}$$

A-12.6 Referee Method

Determine chromium content by atomic absorption spectrophotometer in accordance with method given in IS 13319. Alternatively, it can be analyzed by ICP-OES.

A-13 DETERMINATION OF CADMIUM

Follow the method by AAS as prescribed in IS 3025 (Part 41). Alternatively, it can be analyzed by ICP-OES.

A-14 DETERMINATION OF SELENIUM

Follow the method by AAS as prescribed in IS 3025 (Part 28). Alternatively, it can be analyzed by ICP-OES.

A-15 DETERMINATION OF PHENOLIC COMPOUNDS

A-15.1 Principle

Phenol gives coloured compound when treated with 4-aminophenazone and oxidizing agent, such as, hexacyanoferrate (III) ions in alkaline solution. This is extracted with chloroform and determined quantitatively by spectrophotometry.

A-15.2 Apparatus

A-15.2.1 *Separatory Funnel*, 250 ml

A-15.2.2 *Spectrophotometer*

A-15.2.3 *p*H *Meter*

A-15.3 Reagents

A-15.3.1 *4-Aminophenazone* — Dissolve 3 g of compound in 100 ml of distilled water [Solution A].

A-15.3.2 *Potassium Hexacyanoferrate [III] (Potassium Ferricyanide)* — Dissolve 2 g of the solid in 100 ml of distilled water [Solution B].

A-15.3.3 *Aqueous Ammonia* [4 M]

A-15.4 Procedure

A-15.4.1 Dissolve 1 g of the sample in the distilled water boil, filter and transfer into 100 ml-volumetric flask, make up to the mark, mix thoroughly.

Transfer 10 ml of this solution to a 100 ml-beaker, add 5 ml of citric acid and adjust the *p*H to 9.7-10.3 [use *pH* meter].

Transfer it to a separatory funnel and add 1.0 ml of solution A followed by 10.0 ml of solution B. Shake well to ensure thorough mixing, and then carryout three extractions with successive portion of 15 ml, 10.0 ml and 5 ml of chloroform.

Combine the chloroform extract and make up the volume to 50 ml.

Measure the absorbance of the extract within 10 min [the colour may tend to fade after 10 min] against a blank of chloroform at the wave length of 460 nm. Construct a calibration curve using a series of various concentration of standard phenol solution by treating it in the same way as for the sample solution.

A-15.5 Standard Phenol Solution

A-15.5.1 *Stock Phenol Solution*

Dissolve 1.00 g phenol in 500 ml of distilled water and dilute to 1 000 ml. Add 1 g $CuSO_4$ and 0.5 ml concentrated H_2SO_4 as preservative (1.0 ml = 1.0 mg phenol).

A-15.5.2 *Standard Phenol Solution A*

Dilute 10.0 ml of stock phenol solution to 1 000 ml (1.0 ml = 0.01 mg phenol).

A-15.5.3 *Standard Phenol Solution B*

Dilute 100.0 ml of standard phenol solution A to 1 000 ml with distilled water (1.0 ml = 0.001mg phenol).

A-15.6 Calculation

$$\text{Phenolic compounds (as } C_6H_5OH\text{), ppm} = \frac{\text{Mass of phenol, in microgram} \times 10}{\text{Mass of the sample taken for test, in g}}$$

A-16 DETERMINATION OF ANIONIC DETERGENT

A-16.1 Principle

The aqueous solution of ionic detergent reacts with methylene blue [cationic dye]. Reaction takes place between the ionic dyestuff [which is chloride] and detergent. The reaction product can be extracted by chloroform. The intensity of the colour in the chloroform layer is proportional to the concentration of detergent which can be determined by colorimetric as methylene blue active substance [MBAS].

A-16.2 Reagents

A-16.2.1 *Methylene Blue Solution* — Dissolve 0.1 g of the pure chemical in 100 ml distilled water.

A-16.2.2 *Hydrochloric Acid (6 M)*

A-16.2.3 *Chloroform*

A-16.3 Procedure

A-16.3.1 Weigh 1.0 g of the sample and dissolve it in approximately 25 ml of distilled water boil and filter and transfer to a 100 ml volumetric flask, cool to the room temperature and make to the mark. Mix thoroughly.

Take 25 ml of this solution into a 150 ml-separatory funnel, if required, neutralize it by hydrochloric acid solution. Add 20 ml of chloroform and 1 ml of methylene blue solution. Shake for 1 min, allow to stand for 5 min, then run the chloroform layer into a 100 ml-separatory funnel; retain the aqueous layer (solution A). Add 20 ml of distilled water to the chloroform solution, shake for 1 min, allow to stand for 5 min, then transfer the chloroform layer through a small filter funnel with a cotton-wool plug at the apex, into a 100 ml-volumetric flask. Repeat the extraction of solution A three more times following the above procedure exactly. After the final extraction rinse the filter funnel and plug with a little chloroform, and finally make the solution in the volumetric flask up to the mark with chloroform.

Measure the absorbance of this solution at 650 nm (red filter) against a blank of distilled water.

Plot a graph using a series of standard solution of detergent in place of sample by following the above procedure exactly.

A-16.3.2 *Preparation of Standard Solution*

A-16.3.3 *Stock Linear Alkylbenzene Sulphonate (LAS)*

Weigh accurately 1.0 g of linear alkylbenzene sulphonate (LAS) on a 100 percent basis and dissolve it in water and dilute to 1 000 ml.

1.0 ml = 1.0 mg of LAS

Store the above stock solution in refrigerator to minimize biodegradation. If necessary prepare weekly.

A-16.3.4 *Standard LAS Solution*

Dilute 10 ml of stock LAS solution to 1 000 ml with distilled water.

1 ml = 10 μg LAS

Use above standard solution for the preparation of calibration curve.

A-16.4 Calculation

$$\text{Anionic detergent, ppm [as MBAS; calculated as LAS. Molecular weight (\textit{see} Note)]} = \frac{\text{Mass of the LAS [microgram]} \times 4}{\text{Mass (g) of the sample taken for test}}$$

NOTE — Mention the molecular weight of LAS which is used for the preparation of the standard solution.

ANNEX B

(*Clause* 6)

SAMPLING OF ALUMINO-FERRIC

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling implement and the containers for samples from adventitious contamination.

B-1.2 The samples shall be placed in clean, dry and airtight glass or other suitable containers on which the material has no action.

B-1.3 Each sample container shall be sealed airtight after filling and marked with full details of the material and the date of sampling.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the material of the same grade in a single consignment shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacturer of a batch plant, the material from each batch shall constitute a separate lot. In case of a consignment from a continuously operated plant, a day's (24 h) production shall constitute a lot.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, samples shall be tested from each lot separately. The number of packages or slabs or containers to be selected from lots of different sizes shall be in accordance with Table 2.

Table 2 Number of Packages or Slabs or Containers to be Selected from Lots of Different Sizes

(*Clause* B-2.2)

Sl No. (1)	Lot Size N (2)	Sample size n (3)
i)	Up to 15	3
ii)	16-50	5
iii)	51-100	8
iv)	101 and above	13

B-2.2.1 The package or containers shall be selected at random from the lot. In order to ensure the randomness of selection, random sampling procedures given in IS 4905 may be followed.

B-2.3 Preparation of Individual Test Samples and Composite Samples

B-2.3.1 Draw by any suitable means a representative portion of the material sufficient for carrying out the tests specified in Table 1, from each package or slab or container selected according to **B-2.2**. These portions shall constitute the individual test samples.

B-2.3.2 From each of the individual test samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute the composite sample.

B-2.3.3 The material in the individual test samples and the composite sample shall be transferred to separate containers and labelled with full identification particulars of the samples.

B-2.4 Number of tests

The complete analysis is to be carried out only on composite sample of the lot.

B-2.5 Criteria for Conformity

B-2.5.1 *For Individual Samples*

If all the individual test results for water soluble aluminium compounds and *p*H meet the relevant requirements, the lot shall be deemed satisfactory with regard to these characteristics.

B-2.5.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test results shall satisfy the corresponding requirements specified in Table 1.

B-2.5.3 The lot shall be declared as conforming to the requirements of this specification, if **B-2.5.1** and **B-2.5.2** are satisfied.

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